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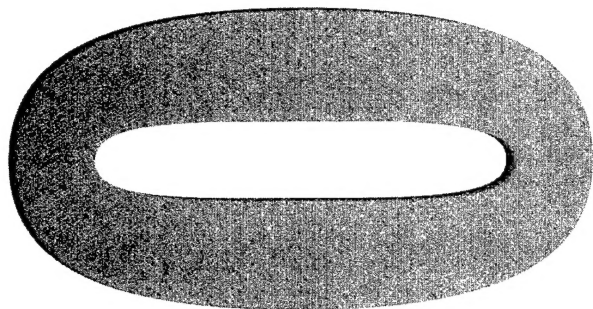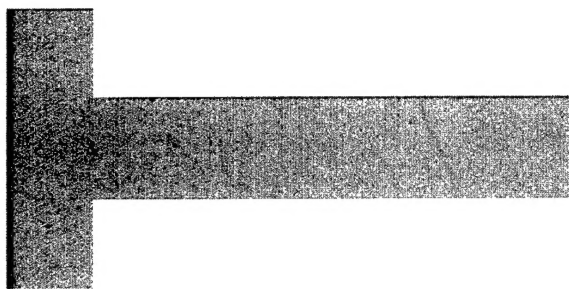
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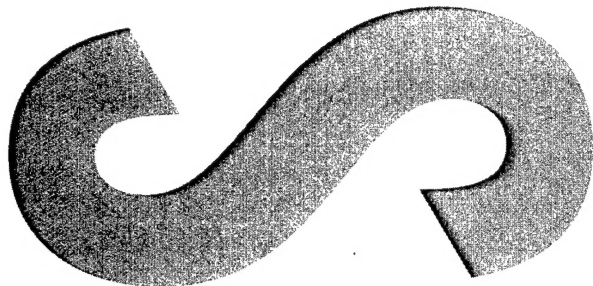
Defence Science and
Technology Organisation

Aug 2003

Evaluation of PBXN-109: the Explosive Fill for the Penguin Anti-Ship Missile Warhead

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DSTO-TR-1471

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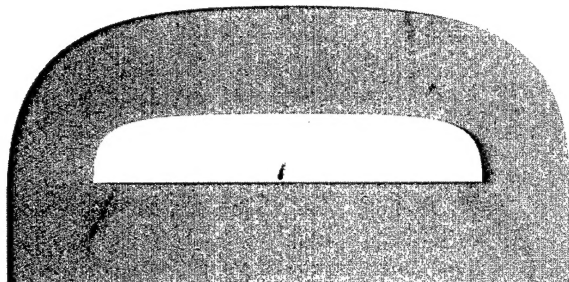# Evaluation of PBXN-109: the Explosive Fill for the Penguin Anti-Ship Missile Warhead

Brian L. Hamshere, Ian J. Lochert and Richard M. Dexter

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ABSTRACT

The RAN has acquired the Penguin Anti-Ship Missile for deployment from Kaman SH-2G(A) Super Seasprite helicopters which are to be carried by the ANZAC frigates. The Penguin manufacturer, Kongsberg Defence and Aerospace of Norway, negotiated an industry off-set program with local industry, a component of which was for ADI Ltd to fill the missile warhead with the polymer bonded explosive PBXN-109. This was the first example of Australian industry production of a PBX for service use. DSTO was tasked to provide appropriate local advice as required to the Penguin Project Office and ADI Ltd during introduction into service and onwards through life. This report details the support to this programme and the the results of work to establish a database on processing, chemical and mechanical properties, performance, and hazards response of PBXN-109.

RELEASE LIMITATION

Approved for public release

AQ F04-03-0214

APPROVED FOR PUBLIC RELEASE

Evaluation of PBXN-109: the Explosive Fill for the Penguin Anti-Ship Missile Warhead

Executive Summary

The Royal Australian Navy has acquired the Penguin Anti-Ship Missile (ASM) for deployment from Kaman SH-2G(A) Super Seasprite helicopters which are to be carried by the ANZAC frigates. The missiles were procured under Project Sea 1414 Phases 1 and 2 while the helicopters are being procured under Project Sea 1411. The Penguin ASM manufacturer, Kongsberg Defence and Aerospace (KDA) of Norway, negotiated an industry off-set program with various Australian industries to provide a local support capability. ADI Ltd was awarded a contract to fabricate parts of the warhead and to fill it with a polymer-bonded explosive, PBXN-109. In addition, other areas of Australian industry were awarded contracts to manufacture the precision flight control surfaces and for the design and manufacture of specialist packaging for the All Up Round and wings and canards. This Australian industry involvement represented 12.4% of the contract.

In keeping with a policy for the Australian Defence Force (ADF) to consider the acquisition of Insensitive Munitions for future weapon systems the explosive chosen for the Penguin ASM warhead was PBXN-109. This polymer bonded explosive was processed and filled into the warhead by ADI Ltd at their Mulwala facility. This was the first example of Australian industry production of a PBX for service use and while ADI was not unfamiliar with the processing of composite energetic materials (they have considerable experience with the composite rocket propellant for Nulka) they had no practical experience with PBXs. Consequently, production was undertaken using a technology transfer package supplied by KDA and utilising fully imported qualified ingredients.

At the commencement of the warhead production programme DSTO was tasked to provide appropriate local advice as required to the Penguin Project Office and ADI Ltd during introduction into service and onwards through life. General areas of concern were identified as initial processing of PBXN-109, chemical and mechanical properties of the resultant charges, performance and generic IM characteristics, and through life support in terms of ageing and the effects of ageing on IM characteristics.

Initial studies were directed at establishing the characteristics of the ingredients and establishing the safety of handling these ingredients through a series of sensitiveness tests. Processing studies were performed to evaluate mixing and casting properties of PBXN-109 and methods of improving them were investigated. The viscosity of the PBXN-109 slurry was shown to be very sensitive to the physical properties of the explosive ingredient, RDX. A comprehensive range of testing of the cured explosive was conducted to establish a database of properties. These tests included sensitiveness

testing, mechanical properties testing and explosive performance tesing. All of the testing produced results in good agreement with published data.

To estimate the response of PBXN-109 to cook-off and shock aggression tests were conducted in laboratory-scale equipment, viz. super small-scale cook-off bomb and large scale gap test. The result of this testing showed PBXN-109 to react relatively mildly during cook-off and to be moderately sensitive to shock, again, in keeping with published data. An ageing trial was conducted to determine whether long-term storage would have a deleterious effect on the mechanical properties of PBXN-109 and whether any adverse changes would be exhibited in shock sensitivity and cook-off tests. While little change was observed in these properties there was a suggestion that the glass transition temperature (T_g) may have increased from approximately $-50°C$ to $-20°C$. As there is a possibility that an increase in T_g may increase shock sensitivity at low temperatures it is recommended that further work be carried out to clarify this point.

As a result of this programme of work a much better understanding of the nature and properties of PBXN-109 has been established and peripheral experiments have resulted in an effective means of improving processing and casting properties, and of reducing the shock sensitivity of PBXN-109. The properties determined across a wide spectrum of tests form the basis of a useful database for future work.

Authors

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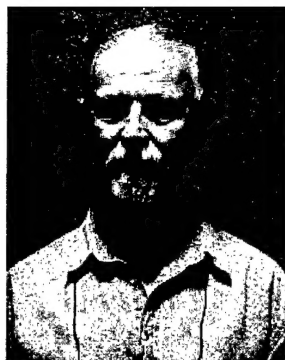
Since 1977 Brian Hamshere has worked in the area of composite energetic materials. Earliest work concentrated on solid composite rocket propelants including high burn rate, low signature and low vulnerability formulations. Considerable effort was expended on developing a plateau burning propellant for the Nulka hovering rocket and assisting in the technology transfer to industry. More recently his attention has been directed towards formulating and developing polymer bonded explosives. Much of this work has centred on the explosive for the Penguin ASM warhead and on investigating explosive formulations for Insensitive Munitions.

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Weapons Systems Division

After completing his PhD in synthetic and physical organic chemistry at Flinders University, Ian spent two years at the University of Melbourne as a post-doctoral fellow where he studied resin derived carbon composites. Since joining the Explosives Group at DSTO in 1998 he has been working in three main research areas: formulation and testing of polymer bonded explosives, the synthesis and characterisation of the insensitive explosive FOX-7, and investigations into Reduced Sensitivity RDX, a grade of RDX with significant potential in the Insensitive Munitions area

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Richard Dexter gained his BSc (Honours) in 1996 from Flinders University. On joining DSTO in 1998 his work included research into PBX formulations, and sensitiveness testing of energetics. In 2000 he was a member of the Scientific Support Group providing scientific advice to the Joint Incidence Response Unit during the Sydney Olympic Games. In 2001 he moved to Land Operations Division where he is now the team leader for the Small Units Combat Simulation CAEn (Close Action Environment). During 2002 he deployed to East Timor as Officer-in-Scientific-Charge of a team studying the impact of new technologies on the operations of the deployed battalion.

Contents

Abbreviations

ADI	ADI Limited
ADF	Australian Defence Force
AO	Antioxidant
ASM	Anti-ship missile
BAM	Bundesanstalt für Materialprüfung
Composition B	RDX/TNT 60/40
CPIA	Chemical Propulsion Information Agency
CXM-7	RDX coated with DOA
DOA	Dioctyl adipate
DSC	Differential scanning calorimetry
DSTO	Defence Science and Technology Organisation
DTA	Differential thermal analysis
ESD	Electrostatic spark discharge
F of I	Figure of insensitiveness
FSSS	Fischer Sub Sieve Sizer
GPa	Gigapascal
HMX	Cyclotetramethylenetetranitramine
HTPB	Hydroxyl terminated polybutadiene
ICRPG	Interagency Chemical Rocket Propulsion Group
id	Inside diameter
IM	Insensitive munitions
IPDI	Isophorone diisocyanate
KDA	Kongsberg Defence and Aerospace
LSGT	Large scale gap test
MPa	Megapascal
MRL	Materials Research Laboratory (DSTO)
NAWC	Naval Air Weapons Center
NOL	Naval Ordnance Laboratory
NWC	Naval Weapons Center
od	Outside diameter
PBX	Polymer (or plastic) bonded explosive
PBXN	PBX formulation qualified for in-service use by the US Navy
RDX	Cyclotrimethylenetrinitramine
RO	Royal Ordnance Plc, Glascoed, UK
SD	Sympathetic Detonation
SEM	Scanning electron microscope
SSCB	Super small-scale cook-off bomb
STDEV	Standard Deviation
T of I	Temperature of ignition
TGA	Thermogravimetric analysis
TMD	Theoretical maximum density
TNT	2,4,6-trinitrotoluene
TPB	Triphenylbismuth
VTS	Vacuum thermal stability
WSD	Weapons Systems Division (DSTO)

1. Introduction

The Royal Australian Navy has acquired the Penguin Anti-Ship Missile (ASM) for deployment from Kaman SH-2G(A) Super Seasprite helicopters which are to be carried by the ANZAC frigates. The missiles were procured under Project Sea 1414 Phases 1 and 2 while the helicopters are being procured under Project Sea 1411. The Penguin ASM manufacturer, Kongsberg Defence and Aerospace (KDA) of Norway, negotiated an industry off-set program with various Australian industries to provide a local support capability. ADI Ltd was awarded a contract to fabricate parts of the warhead and to fill it with a polymer bonded explosive, PBXN-109. In addition, other areas of Australian industry were awarded contracts to manufacture the precision flight control surfaces and for the design and manufacture of specialist packaging for the All Up Round and wings and canards. This Australian industry involvement represents 12.4% of the contract.

The helicopter launched version of the missile is designated Penguin Mk 2 Mod 7 (AGM-119B) and has an operational range in excess of 30 km powered by a two stage solid rocket motor to high subsonic speeds. The inertial navigation system provides the missile with target selection capability and the ability to utilise an operator designated waypoint. A high resolution passive IR seeker provides target selection and acquisition and directs the warhead to a target point at the waterline.

The Penguin ASM warhead consists of a semi-amour piercing steel body 250 mm diameter and 800 mm long developed from an earlier Mk 19 AGM-12 Bullpup missile. The warhead has an all up weight of approximately 120 kg and contains 43 kg of high explosive. The warhead is designed to penetrate the side plate of a ship just above the waterline with sufficient delay that the explosive detonates within the confines of the hull.

In keeping with a policy for the ADF to consider the acquisition of Insensitive Munitions for future weapon systems the explosive chosen for the Penguin ASM warhead was PBXN-109. This polymer-bonded explosive was processed and filled into the warhead by ADI Ltd at their Mulwala facility. This was the first example of Australian industry production of a PBX for service use and while ADI was not unfamiliar with the processing of composite energetic materials (they have considerable experience with the composite rocket propellant for Nulka) they had no practical experience with PBXs. Consequently, production was undertaken using a technology transfer package supplied by KDA and utilising fully imported qualified ingredients.

It should be noted that the Penguin warhead is not a fully IM compliant munition. Testing performed by the US Navy [1, 2] showed that the Penguin warhead met NAVSEAINST 8010.5 IM criteria for fast cook-off, slow cook-off, bullet impact and fragment impact. It also successfully passed the Track Test in which a warhead was accelerated to 950±50 feet per second (290±15 m/s) and impacted into a ship target penetrating the plate with survival of the warhead and its capability of detonating its entire explosive content [1]. However, the Penguin warhead failed to meet Sympathetic Detonation passing criteria for

NAVSEAINST 8010.5 even with shielding [2]. While it was considered that additional work on protective shields, container design and magazine configuration could overcome the problem, a lack of assets, a small inventory of Penguin missiles, and lack of program funds prevented further efforts [2].

At the commencement of the warhead production programme DSTO was tasked to provide appropriate local advice as required to the Penguin Project Office and ADI Ltd during introduction into service and onwards through life. General areas of concern were identified as initial processing of PBXN-109, chemical and mechanical properties of the resultant charges, performance and generic IM characteristics, and through life support in terms of ageing and the effects of ageing on IM characteristics.

The work conducted in support of this programme provides a useful local technology database on processing, chemical and mechanical properties, performance and hazards response of PBXN-109. It also provides through-life support of Penguin ASM in terms of the PBXN-109 warhead filling and the effects of ageing on IM characteristics.

2. Formulation and Ingredients

2.1 PBXN-109 Formulation

The forerunner to the polymer bonded explosive PBXN-109 was developed in the US Navy laboratories at White Oak, Maryland for use as a general purpose bomb fill and was accepted for in-service use during the late 1980s. The military specification, MIL-E-82886(OS) [3], details the formulation and quality assurance testing of this explosive.

Table 1 PBXN-109 Formulation

Ingredient	Nominal Weight %
RDX	64.00
Aluminium	20.00
Hydroxyl-terminated polybutadiene (HTPB)	7.346
Dioctyl adipate (DOA)	7.346
Antioxidant 2246 (AO)	0.10
N,N'-di(2-hydroxyethyl) dimethylhydantoin (Dantocol DHE)	0.26
Triphenylbismuth (TPB)	0.02
Isophorone diisocyanate (IPDI)	0.95

A more detailed description of the formulation is given in MIL-E-82886(OS) [3] and in Section 2.2.

2.2 Ingredients

At the commencement of this program of work it was deemed essential that all PBXN-109 processing and testing conducted at DSTO must be carried out using PBXN-109 made with ingredients from the same lots as used by ADI to manufacture the warheads. All ingredients procured by ADI were imported and fully qualified for the production of PBXN-109. DSTO was given an allocation of those ingredients sufficient to produce approximately 200 kg of PBXN-109.

The RDX used for the production of PBXN-109 was manufactured by Dyno Nobel, Norway and supplied as CXM-7 [4]. CXM-7 consists of RDX that has been coated with dioctyl adipate (DOA), the same plasticiser as used in the PBXN-109 formulation, to desensitise the RDX for handling purposes. The composition of CXM-7 as specified in [4] is reproduced below.

Table 2 Composition of CXM-7

Component	Weight, %	Specification
Dioctyl adipate, DOA	4.0 to 5.5	DOD-D-23443
RDX, Type II, Class 1 plus RDX, Type II, Class 5	94.5 to 96.0	MIL-DTL-398D Nominal* or specification grade

* Nominal grade refers to RDX made in accordance with the respective process for producing the specific class but is not inspected for granulation requirements.

WS26702 [4] states that "the ratio of RDX, Type II, Class 1, to RDX, Type II, Class 5, shall be 95 to 5 based on input weights".

The process that yields Type II RDX also produces HMX as a by-product and consequently the CXM-7 contains from 5 to 12% of this material.

The aluminium manufactured by Toyal America, Inc. is designated ATA X-81 Aluminium Powder and is reported as having an average particle size between 12.0 and 18.0 microns (determined by FSSS technique). The material complies with MIL-A-23950A (Amendment 1), Type IV Specifications.

The binder ingredients HTPB R45HT, DOA, AO (2,2'-methylene-bis[6-tertbutyl-4-methylphenol]), Dantocol DHE (N,N'-di(2-hydroxyethyl) dimethylhydantoin), triphenylbismuth (cure catalyst) and IPDI (curing agent) all complied with specification limits as tested by the ADI laboratory.

The exact composition of the PBXN-109 formulation to be processed is dependent upon the equivalent weights of the reacting binder ingredients (HTPB, Dantocol DHE and IPDI)

and on the amount of DOA in the CXM-7. Adjustments are made to the formulation whenever there is a change to these properties due to the use of a different batch or lot of the material. The formulation is determined using an isocyanate to hydroxyl functional group molar ratio of 1.

In addition to the ingredients required to make up the explosive composition there also are two polyolefin wax-like materials used to coat the inner surface of the warhead to give added protection to the explosive fill. The liner material is applied as a hot melt consisting of 20% Epolene Wax and 80% Rextac.

3. Results - Processing and Testing

3.1 Characterisation of Ingredients

The ingredients received at DSTO Edinburgh were subjected to a series of tests to assess that they were safe to handle and to process and were compatible when incorporated in the explosive formulation. These tests included sensitiveness testing (Rotter Impact (F of I), BAM Friction, Temperature of Ignition (T of I), Electrostatic Discharge (ESD) and Vacuum Thermal Stability (VTS)), particle size determinations and scanning electron microscopy (SEM) of the solid ingredients (RDX and aluminium).

3.1.1 Sensitiveness Testing

A summary of the data from the series of sensitiveness tests and vacuum thermal stability tests for the ingredients and PBXN-109 is presented in Table 3 as the average result from three batches.

Table 3 Sensitiveness Testing

Test	CXM-7	CXM-7 (no DOA)	PBXN-109	PBXN-109/Liner
F of I[1]	170 (12.7)	80 (nr)	140-180 (3.3)	-
BAM Friction (N)	192	108	252 - >360	-
T of I (°C)	214	213	221	-
ESD - ignition (J)	4.5	4.5	-	-
ESD - no ignition (J)	0.45	0.45	4.5	-
VTS (mL/g)	0.08	0.03	0.03	0.08

[1] average volume of gas evolved, mL quoted in parentheses (nr - not recorded)

3.1.2 Particle Size

The particle sizes of the two solid ingredients, RDX and aluminium, were determined using a Malvern Mastersizer 2000. The DOA coating on the RDX was removed by solvent wash prior to measurement. In Figure 1 the particle size distribution curve for the RDX shows a broadening at the low particle size edge of the curve that may be

attributed to the presence of a small quantity of RDX Class 5. The particle size distribution for the aluminium X-81 is shown in Figure 2, the small peak between 200-300 µm is thought to be due to a small number of agglomerates. The particle size details for both ingredients are presented in Table 4. The large value recorded for the span of both materials is indicative of a wide range of particle sizes within the distribution.
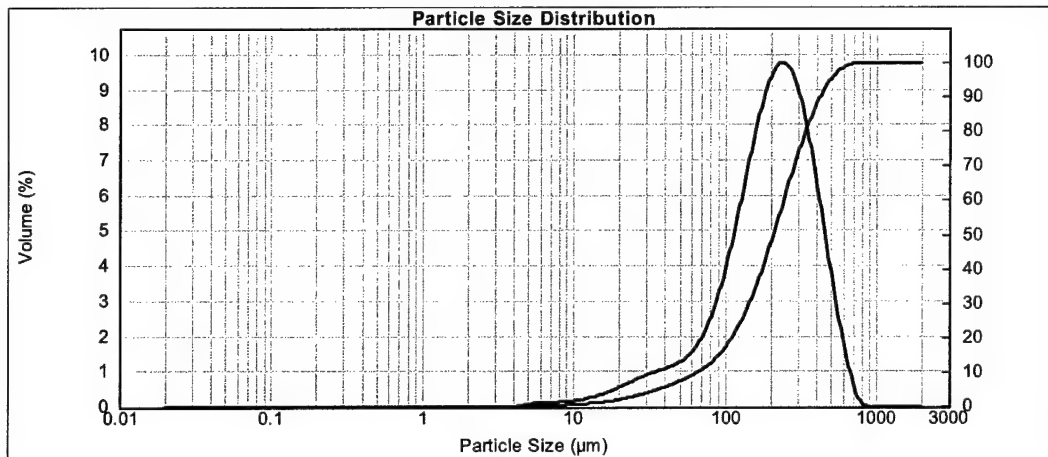
Figure 1 *Particle size distribution for RDX from CXM-7*
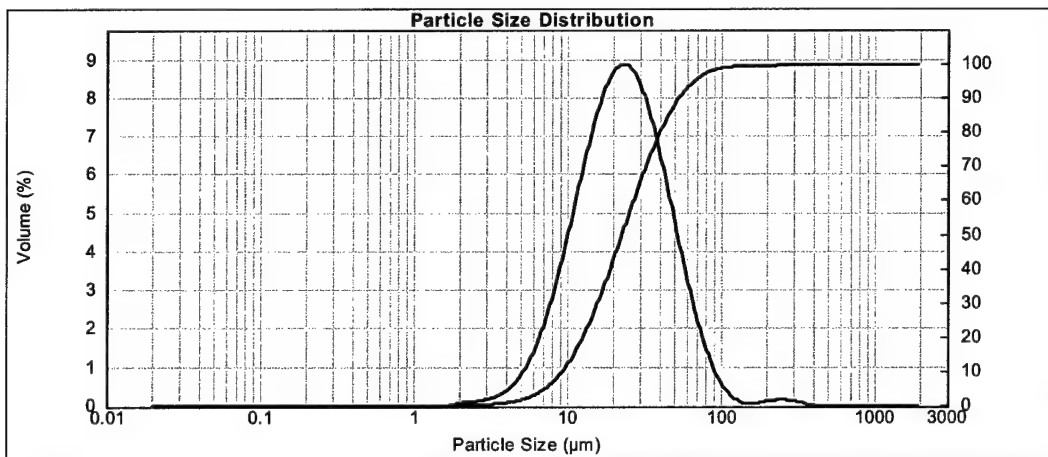
Figure 2 *Particle size distribution for aluminium powder, X-81*

Table 4 Particle Size Data

	Particle Size Distribution			
	d(0.1) μm	d(0.5) μm	d(0.9) μm	Span$^\Psi$
RDX (CXM-7)	67	208	424	1.7
Aluminium (X-81)	9	23	54	1.9

$^\Psi$ Span = (d(0.9)-d(0.1))/d(0.5)

3.1.3 Scanning Electron Microscopy

The RDX and aluminium powders were examined by scanning electron microscopy (SEM), and typical images are presented in Figures 3 & 4 and 5 & 6 respectively. The RDX crystal morphology shows a distinct angular appearance with sharp features. There is also a considerable amount of smaller irregular shaped particles suggesting the RDX may have been subjected to some degree of grinding (Fig. 3 & 4).

Figure 3 SEM image of CXM-7

Figure 4 SEM image of CXM-7

The apparent particle size as determined from the magnified images is consistent with the particle size determined by the light scattering technique shown in Table 4. The aluminium particles are essentially spheroidal in shape and while there is a considerable range in size (Fig. 5 & 6) the results are consistent with those presented in Table 4.

Figure 5 SEM image of Al X-81
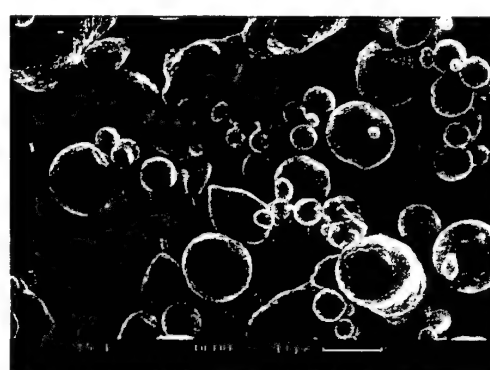
Figure 6 SEM image of Al X-81

3.1.4 Liner Material

Although not an ingredient of the explosive formulation, the liner material separates PBXN-109 from the internal steel wall of the warhead and is thus in intimate contact with the explosive. While the main interest concerns the chemical compatibility of the liner with the explosive, additional tests were performed to establish the thermal properties of the liner. These tests included DSC, DTA and TGA and are included with VTS and density results in Table 5.

Table 5 Properties of liner material

	Rextac	Epolene N-10	Rextac + Epolene	Rextac/Epolene + PBXN-109
VTS	0.14 mL.g^{-1}	0.07 mL.g^{-1}	0.12 mL.g^{-1}	0.08 mL.g^{-1}
Density	0.87 g.cm^{-3}	0.92 g.cm^{-3}	0.89 g.cm^{-3}	-
DSC				
Onset of decomposition	225°C	225°C	240°C	-
TGA				
Onset of wt loss	50°C	275°C	275°C	-
DTA Onset	380°C	240°C	250°C	-
Major peak	410°C	325°C	330°C	-

3.2 Processing Studies

The initial mixes of PBXN-109 were prepared in 500 g quantities using a one pint Baker Perkins planetary action mixer. These mixes provided sufficient material for assessing sensitiveness and processing properties of PBXN-109. As the requirement for larger quantities of explosive for performance testing and ageing studies arose, 2 and 5 gallon mixers, also planetary action, were used. All mixes were conducted under vacuum (<10 torr) at a temperature of 60°C.

3.2.1 Mixing Procedure

The mixes were prepared according to conventional practice, the binder precursors were thoroughly mixed and degassed before the solid ingredients - aluminium powder and then the RDX (as CXM-7), were added. The last step of the mixing operation was the addition of the diisocyanate curing agent, IPDI, which was mixed for a short duration prior to casting the PBX into a mould. The details of the mix procedure are outlined in Table 6 below.

Table 6 PBXN-109 mix procedure

Ingredients/Action	Mix time (no vacuum) Minutes	Mix time (vacuum) Minutes
HTPB + DOA + Dantocol + AO + TPB	2	28
Aluminium	2	13
CXM-7 (50%)	2	13
CXM-7 (25%)	2	13
CXM-7 (25%)	2	13
Scrape down	-	-
Mix	0	60
IPDI	1	4
Scrape down	-	-
Mix	0	15

During the course of investigations into the effects of varying the relative amounts of Class 1 and Class 5 RDX on the processing properties of the PBXN-109 formulation a variation to the above procedure was made. A master batch of the binder ingredients was used to expedite the mixing procedure and to ensure uniformity of composition across a range of small-scale (500 g) mixes. While using this technique it was discovered that the cure catalyst, triphenylbismuth, must not be included in the master batch of binder but should be added at the time of the mix. Incorporation of the TPB in the master batch greatly accelerates the cure reaction resulting in an unacceptable pot life for the PBX, i.e. the length of time that the PBX remains in castable condition after the addition of the curing agent, IPDI. This effect is illustrated in Section 3.2.3.

3.2.2 Casting

At the completion of the mixing cycle the PBXN-109 slurry was vacuum cast into moulds appropriate for the testing required. During this operation vibration was applied to the entire assembly to facilitate degassing. Where multiple moulds were to be filled (accommodated on a carousel within the casting box) a sequential filling operation was adopted and the vacuum was released and re-applied several times during filling to assist in the removal of trapped air pockets. The casting duration varied depending on the size and number of items being filled in an operation and was typically from 10 to 30 minutes. Where multiple test pieces or charges were being filled two or three operations were sometimes necessary.

3.2.3 Viscosity

To quantify the processing properties of the PBXN-109 slurry it was planned to determine its viscosity as a function of time while subjected to a constant shear rate. The Haake VT 550 viscometer was to be employed for this purpose but, because of the viscous nature of PBXN-109 and the limitations of the viscometer, meaningful data could not be obtained. However, during investigations into the effect on viscosity of varying the RDX particle size distribution the viscometer proved a valuable tool. The instrument was run at a constant rotational speed of approximately 1 rpm giving a shear rate of approximately 0.7/s when using a SVP II sensor system, the test sample was maintained at 60°C.

In this study the CXM-7 was replaced with RDX Grade A, Class 1 (coarse) and RDX Class 5 (fine) in various ratios and is more fully covered in a separate report [5]. Typical results are shown in Figure 7 for PBXN-109 formulations prepared from a master batch of binder pre-blend containing the catalyst TPB.

Viscosity measurements were also compared for similar batches of PBX that were formulated with a master batch of binder. In the first batch the cure catalyst, TPB, was included in the master batch pre-blend while in the second the cure catalyst was omitted but added at the start of the mix cycle. The viscosity curves for the two formulations (PBXN-109 with 54% RDX Grade A, Class 1 and 10% RDX Class 5) showed that the master batch of binder was strongly catalysed by the pre-addition of the TPB resulting in a PBX with a pot life of less than 20 minutes. The second formulation, in which the catalyst was added at the start of the mix cycle, displayed a very slow initial rate of cure having attained a viscosity of 300 Pa.s after 3 hours 50 minutes (not shown in Fig. 7).
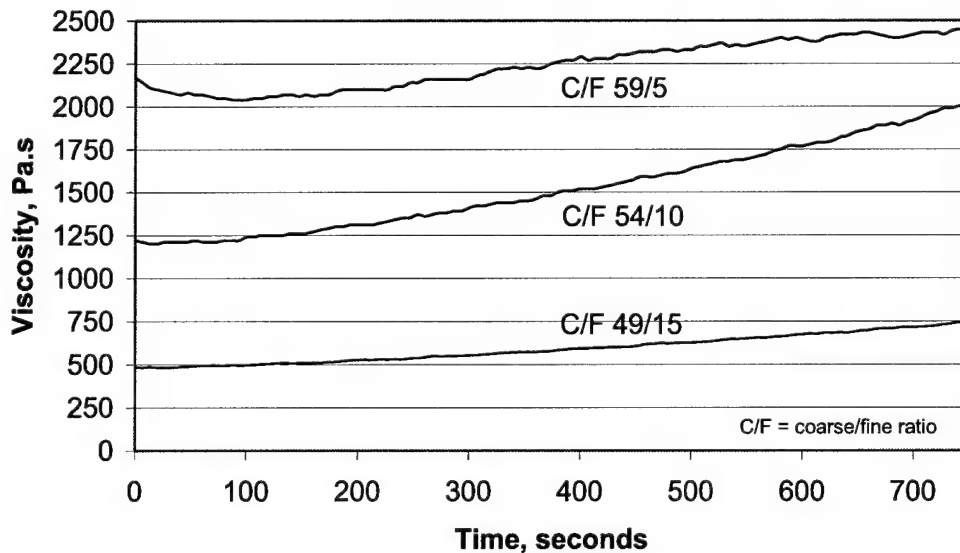

Figure 7 Effect of RDX particle size distribution on viscosity of PBXN-109

During the course of this work the manufacturer of the CXM-7 (Dyno Nobel) supplied ADI Ltd with a sample quantity of a modified version of CXM-7 that they claimed would enhance the processing properties of PBXN-109. This material was provided to DSTO for evaluation. While the material did not meet all the claims made of it, it did contribute to PBXN-109 with superior processing properties due to the more rounded nature of the RDX particles. The outcomes from the DSTO evaluation of the new CXM-7 have been reported separately [6].

3.3 Characterisation of PBXN-109

In addition to the characterisation of the ingredients used in PBXN-109, tests were performed on the cured explosive to establish its hazards sensitiveness properties such as response to impact, friction, heat and electrostatic discharge. Additional tests were conducted to determine density, and tensile mechanical properties were measured as a function of temperature.

3.3.1 Sensitiveness Testing

The same range of sensitiveness testing as performed on the ingredients was conducted on PBXN-109 and compatibility of the wax liner with the PBXN-109 was evaluated by means of Vacuum Thermal Testing. The results are presented together with those for the ingredients in Table 3.

3.3.2 Density

The density of the cured PBXN-109 was determined using a Micromeritics AccuPyc 1330 gas displacement pycnometer using helium as the working gas. A series of ten measurements was made on each sample and the density expressed as the average value. In addition to measuring the density of the PBX from a number of different batches it was also determined as a function of depth within a vertical casting 50 mm diameter by 300 mm long.

Results for the various batches of PBXN-109 were in the range 1.65 to 1.68 $g.cm^{-3}$ while the densities of samples from 6 equi-spaced samples along the length of the cylindrical charge showed surprising uniformity, 1.656±0.005 $g.cm^{-3}$.

The theoretical maximum density (TMD) was calculated as 1.673 $g.cm^{-3}$ from the component densities 0.93 $g.cm^{-3}$ (cured binder, 16 wt %), 1.82 $g.cm^{-3}$ (RDX, 64 wt %) and 2.70 $g.cm^{-3}$ (aluminium, 20 wt %). The value of the density for the binder was measured from a block of the cured rubber whereas the other two values were taken from the literature. The measured density of PBXN-109 (1.656 $g.cm^{-3}$) is 98.98% TMD.

3.3.3 Plasticiser Loss

To determine whether plasticiser loss from the PBX through volatility was a potential problem an isothermal weight loss experiment was devised. An isothermal thermogravimetric analysis technique was discounted on the grounds of the very small sample size, the irregular sample shape and uncertain surface area to volume ratio. The technique adopted involved filling small aluminium boxes of various dimensions with the PBX such that only one surface was exposed to the atmosphere. By this means the exposed surface area and the depth of the block were accurately known. The samples were held in a 60°C oven and were withdrawn periodically to be weighed.

The samples were maintained at 60°C for 6 months and after 9 weeks a slight wetting of the surface was observed. However, this material did not evaporate and no weight change was recorded for any of the samples. The material was tacky to touch but could not be wiped from the surface of the PBX.

3.3.4 Mechanical Properties

Constant strain rate uniaxial tensile testing of cured samples of PBXN-109 was performed on an Instron 5500R1185 Universal Test Machine. All testing was conducted at a cross-head rate of 50 mm.min^{-1} on JANNAF stamped samples. The machine was fitted with a temperature conditioning cabinet enabling samples to be tested over the temperature range –70°C to 60°C. An extensometer was not used for the testing and all strain measurements were calculated assuming a 69 mm gauge length.

Samples of PBXN-109 were tested from a number of castings with the majority of the tests performed at room temperature, approximately 23°C. To ensure the binder levels were in accordance with the specification a mix was prepared using CXM-7 that had been stripped of its DOA coating. Accurate amounts of the binder materials, particularly the DOA, were added to the formulation. The mechanical properties of this formulation are compared with a standard mix in Table 7. Both mixes were prepared as 500g batches in a 1 pint Baker Perkins mixer. The mechanical properties of PBXN-109 prepared in different capacity mixers are included in the table to identify any scale-up effects. The data presented for a 160 kg batch from a 30 gallon mixer was prepared at ADI – Mulwala (Lot No. PLM99/03) and tested at DSTO.

Table 7 Tensile properties of PBXN-109 at ambient conditions

Property	1 pint (0.5 kg)		2 gal.	5 gal.	30 gal.
	Std mix	CXM-7 DOA removed	6 kg	35 kg	160 kg
Stress (max), MPa	0.61	0.70	0.66	0.68	0.69
Strain at max stress, %	10.7	11.3	11.8	14.6	18.8
Modulus of Elasticity, MPa	8.7	9.48	9.1	8.2	6.5

Additional tests were performed on tensile samples taken from deep (~150 mm) castings to determine the extent of mechanical property variation as a function of depth. Slabs were sequentially cut from the block and numbered 1 from the top through to 7 at the base of the block. The average tensile properties and percentage standard deviations from the three specimens from each slab are presented against slab position in Table 8. At the bottom of the table the average value for all data and their associated percentage standard deviations are also presented.

Table 8 Tensile properties at ambient conditions as a function of position in a deep casting

Slab		Stress (max)		Strain at max stress		Modulus of elasticity	
		MPa	% STDEV	%	% STDEV	MPa	% STDEV
1	Top	0.67	0.88	15.6	6.60	7.70	4.47
2		0.65	0.69	14.7	7.71	7.58	4.49
3		0.66	2.45	15.2	0.82	7.58	3.02
4		0.66	1.98	14.3	1.51	8.14	2.00
5		0.65	0.85	13.3	2.57	8.46	1.50
6		0.64	0.09	12.4	1.80	9.06	2.14
7	Bottom	0.64	1.47	11.5	3.04	9.64	2.51
Average all slabs		0.65	1.96	13.9	10.99	8.31	9.42

Attempts to determine tensile properties as a function of temperature were delayed by equipment failure and a lengthy wait for a replacement conditioning cabinet. The test material was wrapped in aluminium foil and stored at room temperature before being machined into slabs from which the test specimens were stamped. The PBXN-109 was 12 months old at the time of the test. The test specimens for all tests except those conducted at room temperature (23°C) were taken from a single large block of PBXN-109. The 23°C test specimens (6) came from the two central slabs of a similar large block cast from the same batch of PBXN-109 (Table 9). The percentage standard deviations for the properties at each test temperature are shown in brackets in Table 9.

Table 9 Tensile properties as a function of temperature

Test temp	Slab	Stress (max)		Strain at max stress		Modulus of elasticity	
°C	(no. of tests)	MPa	% STDEV	%	% STDEV	MPa	% STDEV
-70	1 (2) Top	2.22	0.92	5.0	4.01	95.3	2.85
-60	2 (3)	1.69	2.36	20.6	17.22	52.5	4.39
-40	3 (3)	1.23	1.82	24.4	3.68	26.0	11.00
-20	4 (3)	0.94	0.66	17.4	5.32	17.3	9.08
0	5 (3)	0.79	0.83	19.4	8.52	9.50	4.38
40	6 (3)	0.63	0.65	14.0	2.09	7.18	2.22
60	7 (3) Bottom	0.57	2.63	13.7	0.28	6.10	2.14
23	- (6) Centre	0.67	1.76	15.50	4.07	7.60	4.21

An attempt was made to determine the bond strength of the PBXN-109 to the wax liner. The Bond-in-Tension test [7] was used in which liner material was coated onto the anvil and PBXN-109 was cast onto the liner surface through the pipe. The technique proved unsatisfactory and no meaningful results were obtained. However, it was evident that the bond strength between the two materials was extremely low.

3.4 Explosive Properties

All detonation experiments were performed on charges boosted with 50:50 pentolite cylinders (length/diameter=1) and initiated with Risi RP-501 EBW detonators. Densities of the charges were 1.65 ± 0.01 g.cm^{-3}.

3.4.1 Velocity of Detonation

The velocities of detonation for charges confined in heavy walled seamless steel tube were determined at three diameters (51, 76 and 102 mm) using coaxial probes (10 per charge spaced at 20.0 mm intervals). The velocities of detonation for unconfined charges were determined at six different diameters (10, 15, 20, 35, 50 and 82 mm) by either digital streak photography or time-of-arrival piezoelectric pins spaced at 20.0 mm intervals along the length of the charge.

Table 10 Velocity of Detonation Data

Dia.(mm)	Confined	Technique	VoD[1] (m.s⁻¹)
51	Yes	Coaxial probes	7527
76	Yes	Coaxial probes	7567
102	Yes	Coaxial probes	7579
10	No	Digital streak imaging	7092
15	No	Digital streak imaging	6862
20	No	Digital streak imaging	7391
35	No	Digital streak imaging	7577
50	No	Digital streak imaging	7678
82	No	Digital streak imaging	7567
82	No	Piezoelectric pins	7617[2]

[1] average from three firings

[2] one firing only

Due to problems with the calibration software for the digital camera the velocity of detonation results measured with this technique are not extremely accurate as is observed for the results at 50 mm diameter, which is unrealistically high.

3.4.2 Detonation Pressure

Relative detonation pressures were determined at two diameters (50 and 82 mm) using the dent test technique [8, 9]. The unconfined charges were detonated on top of a stack of at least three 50 mm thick plates of 250 grade steel with Rockwell hardness B74-76. The dent depths were compared to dents produced by TNT and/or Composition B charges of the same diameters.

Table 11 Relative Detonation Pressure Data for PBXN-109

Diameter mm	Dent depth mm	Detonation pressure (GPa) relative to	
		TNT	Comp B
50	7.78	20.1	19.4
82	14.28	-	18.3

3.4.3 Critical Diameter

The critical diameter was estimated by firing cylindrical charges of various diameters. Limitations of the casting technique prevented determinations below a charge diameter of 10 mm. Success or failure of the charge to sustain a detonation was determined from the digital streak images. As observed from the results presented in Table 10 detonation was sustained at 10 mm charge diameter and the critical diameter is thus less than 10 mm.

3.5 Insensitive Munitions Characterisation

Due to resource limitations only two laboratory scale tests were chosen to assess the IM characteristics of PBXN-109: response to cook-off and shock. The Super Small-scale Cook-off Bomb (SSCB) [10] was used to assess response to fast and slow cook-off and the MRL Large Scale Gap Test (LSGT) [11] was chosen to assess shock sensitivity.

3.5.1 Cook-off Testing

This test is based on the test procedure that was established in 1989 [10] and has since been modified to ensure symmetry at both ends of the test vessel, Figure 8. In addition to determining the temperature of reaction the SSCB test provides an indication of the severity of the reaction for an explosive subjected to a particular heating rate. The heating rate is controlled by the voltage supplied to the heating bands, the slow rate uses a voltage of 120 volts and has an average heating rate of $0.12°C.s^{-1}$, the fast rate uses a voltage of 240 volts and has an average heating rate of $0.76°C.s^{-1}$. A calibrated thermocouple indicates the temperature at which a reaction is initiated while the post-test condition of the vessel determines the severity of the reaction (deformation, size/number of fragments, holes in end plates). The severity of the reaction is described by the following classifications: mild burn, burn, deflagration, explosion, or detonation.
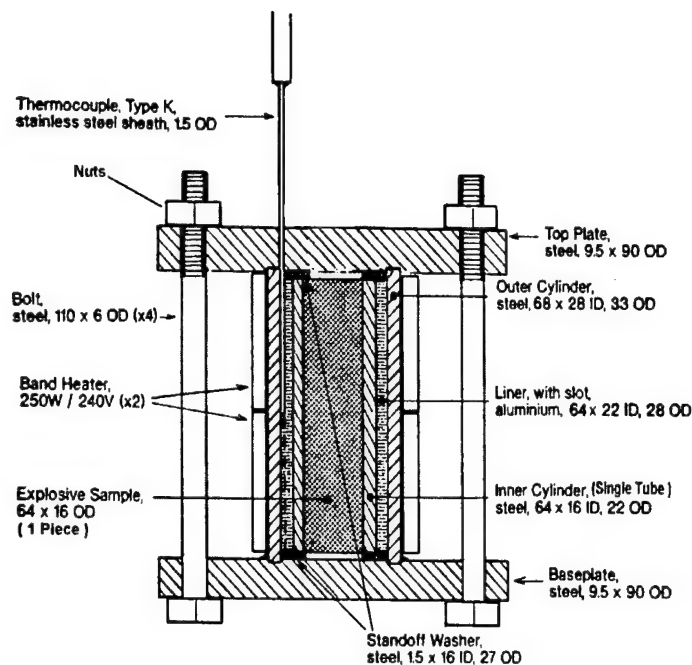
Figure 8 Super Small-scale Cook-off Bomb Test Assembly

The test specimens were prepared by casting the PBXN-109 directly into steel tubes (64 x 22 od x 16 id mm). A duplicate set of specimens was prepared in steel tubes that had been lined with a thin layer of the waxy liner material (Epolene/Rextac) used in the warhead. The results showing temperature of reaction and severity of reaction are presented in Table 12.

Table 12 Fast and Slow cook-off Data

Unlined tubes				Lined tubes			
Fast		Slow		Fast		Slow	
Temp. °C	Event	Temp. °C	Event	Temp. °C	Event	Temp. °C	Event
167	Burn	196	Burn	207	Burn	209	Burn
211	Burn	196	Burn	216	Burn	198	Burn
209	Burn	205	Burn	-	-	207	Burn
196 (av)		199 (av)		212 (av)		205 (av)	

3.5.2 Shock Sensitivity Testing

The shock sensitivity of PBXN-109 was determined using the MRL Large Scale Gap Test [11], a method closely resembling that developed at Naval Ordnance Laboratory. The test was used to determine the shock pressure required to give a 50% probability of detonating the PBXN-109 test sample. The PBXN-109 was directly cast into the LSGT tubes (140 x 48.3 od x 38.1 id mm) and, as for the SSCB test, an additional batch was prepared in tubes that had been lined with the Epolene/Rextac liner. The results from the LSGT are presented in Table 13.

Table 13 Large Scale Gap Test Results

50% Point	Unlined tubes	Lined tubes
Number of cards	196	196
Pressure (GPa)	2.35	2.35

3.6 Ageing Characteristics

The main structural component of PBXN-109 is the plasticised polybutadiene-polyurethane binder made from the primary component ingredients HTPB, DOA and IPDI. It is reasonable to expect that, with time, the chemical properties of the binder may change and that these changes may affect the mechanical properties and possibly the way in which the PBX responds to inadvertent stimuli. To assess the affect of accelerated ageing the mechanical properties of the PBX were determined as a function of time and the affect on cook-off and shock sensitivity responses were determined using SSCB and LSGT tests respectively.

3.6.1 Mechanical Properties

The ageing programme was limited to a comparison of the mechanical properties of PBXN-109 that had been aged at room temperature and at 60°C. The explosive was stored at both temperatures as 25 mm thick slabs (exposed storage) and as 150 mm thick blocks wrapped in aluminium foil (sealed storage). The room temperature storage was not controlled but subject to the seasonal fluctuations experienced in the magazine. The 60°C storage was carried out in water-jacketed ovens. No attempt was made to control or monitor the relative humidity. The testing was carried out in the same manner as described in Section 3.3.4. Equipment problems precluded the determination of mechanical properties after 6 months storage, however, the results for zero storage time and for 12 months storage time at ambient and 60°C for the exposed and sealed explosive are presented below in Table 14. The data presented in Table 14 represents the average mechanical property value from the number of test specimens shown in brackets in the first column. The properties determined from batches of 21 test specimens were taken

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from single larges blocks of PBX. The smaller number of test specimens (3 or 6) were taken from one or two slabs respectively from a large block.

Table 14 The mechanical properties of aged PBXN-109

	Stress (max)		Strain at max stress		Modulus of elasticity	
	MPa	% STDEV	%	% STDEV	MPa	% STDEV
Storage at RT						
Zero time (21)	0.65	1.96	13.9	10.99	8.3	9.42
12 mths-exposed (3)	0.68	0.94	15.1	3.46	8.3	4.36
12 mths-sealed (6)	0.67	1.76	15.5	4.07	7.6	4.21
Storage at 60°C						
Zero time (21)	0.65	1.96	13.9	10.99	8.3	9.42
12 mths-exposed (21)	0.71	1.49	14.2	4.72	9.2	5.50
12 mths-sealed (21)	0.71	1.86	15.0	9.52	8.5	11.09

With spare blocks remaining (unused 6 months storage test material) the mechanical properties as a function of test temperature were determined for PBXN-109 stored for 12 months in the sealed condition at ambient temperature and 60°C. Limited data was also obtained for material subjected to 12 months exposed storage at ambient temperature. The data presented below in Table 15 represents the average value of three test specimens tested at each test temperature apart from those determined at 23°C for which six specimens were used.

Table 15 Mechanical properties of aged PBXN-109 as a function of temperature

Test temperature	Stress (max)		Strain at max stress		Modulus of elasticity	
°C	MPa	% STDEV	%	% STDEV	MPa	% STDEV
12 m at RT 'sealed'						
-70	2.22	0.92	5.01	4.01	95.26	2.85
-60	1.69	2.36	20.60	17.22	52.49	4.39
-40	1.20	1.82	24.84	3.68	30.90	11.00
-20	0.94	0.66	17.35	5.32	17.31	9.08
0	0.79	0.83	19.42	8.52	9.51	4.38
23	0.67	1.76	15.52	4.07	7.62	4.21
40	0.63	0.65	14.03	2.09	7.18	2.22
60	0.57	2.63	13.70	0.28	6.06	2.14

Table 15 (cont.) Mechanical properties of aged PBXN-109 as a function of temperature

Test temperature	Stress (max)		Strain at max stress		Modulus of elasticity	
°C	MPa	% STDEV	%	% STDEV	MPa	% STDEV
12 m at 60°C 'sealed'						
-60	1.92	12.97	5.46	3.43	84.79	8.17
-40	1.24	2.60	6.95	1.37	49.70	4.26
-20	0.97	2.29	14.90	10.45	20.54	12.74
0	0.82	0.77	15.77	3.91	11.61	11.44
23	0.71	1.86	14.95	9.52	8.54	11.09
40	0.65	2.15	13.99	2.93	7.48	3.79
60	0.58	3.84	11.70	8.72	7.23	3.93
12 m at RT 'exposed'						
-40	1.20	0.42	27.17	7.39	30.95	29.07
-20	0.91	0.88	20.13	0.71	19.70	5.99
0	0.77	1.36	16.33	0.33	11.70	4.86
23	0.68	0.94	15.13	3.46	8.30	4.36
60	0.56	0.37	12.18	3.17	6.93	1.68

3.6.2 IM Properties

The affect of accelerated ageing on the shock sensitivity and cook-off properties of PBXN-109 was determined using the LSGT and SSCB tests as described in sections 3.5.1 and 3.5.2 respectively. Test specimens were prepared by casting PBXN-109 into steel LSGT and SSCB tubes some of which had their internal surface lined with wax and the others left as unlined steel. The tubes were divided into batches and stored at either ambient conditions in a magazine or at 60°C in a water-jacketed oven. The test schedule called for samples to be removed and tested after 6 months and 12 months storage.

The results of the cook-off ageing trial showing temperature at reaction and the severity of reaction for samples in lined and unlined tubes that were subjected to fast and slow heating rates are presented below in Table 16 (ambient storage) and Table 17 (60°C storage). NB the zero ageing time reference data has been taken from Table 12.

Table 16 SSCB data for PBXN-109 aged under ambient conditions

Ageing time months	Tube treatment	Temperature at reaction, °C	
		Fast heating rate[Ψ]	Slow heating rate[Ψ]
0	Unlined	167, 211, 209 **[196]**	196, 196, 205 **[199]**
	Lined	207, 216, - **[212]**	209, 198, 207 **[205]**
6	Unlined	178, 169, 211 **[186]**	204, 205, 203 **[204]**
	Lined	204, 169, 185 **[186]**	207, 198, 209 **[205]**
12	Unlined	199, 200, 211 **[195]**	201, 207, 204 **[204]**
	Lined	193, 208, 211 **[204]**	205, 207, 206 **[206]**

[Ψ]Average given in brackets

The reaction severity for all tests was evaluated as a burn.

Table 17 SSCB data for PBXN-109 aged at 60°C

Ageing time months	Tube treatment	Temperature at reaction, °C	
		Fast heating rate[Ψ]	Slow heating rate[Ψ]
0	Unlined	167, 211, 209 **[196]**	196, 196, 205 **[199]**
	Lined	207, 216, - **[212]**	209, 198, 207 **[205]**
6	Unlined	206, 186, 195 **[196]**	210, 195, 206 **[204]**
	Lined	185, 194, 200 **[193]**	203, 212, 202 **[206]**
12	Unlined	224, 207, 207 **[212]**	207, 207, 207 **[207]**
	Lined	205, 218, 202 **[208]**	206, 191, 206 **[201]**

[Ψ]Average given in brackets

The reaction severity for all tests was evaluated as a burn.

The classification of reaction severity was determined from an examination of post cook-off test debris. The extent of damage typical of burn type reactions is shown in Figures 9 and 10.
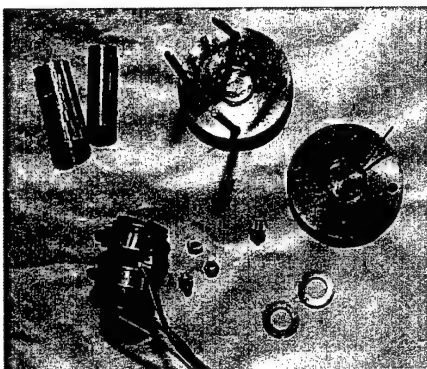
Figure 9 SSCB test debris - burn reaction

Figure 10 SSCB test debris - burn reaction (slightly more severe)

The results of ageing on the shock sensitivity of PBXN-109 are shown in the LSGT data presented in Table 18.

Table 18 LSGT data for aged PBXN-109

Age time months	Ageing environment	Tube treatment	50% Gap No. cards	Pressure GPa
0	-	Lined	196	2.35
0	-	Unlined	196	2.35
6	RT	Lined	198	2.31
6	60°C	Lined	201	2.25
6	60°C	Unlined	198	2.31
12	RT	Lined	195	2.37
12	60°C	Lined	192	2.43
12	60°C	Unlined	195	2.37

4. Discussion

4.1 Characterisation of Ingredients

Characterisation of the ingredient and liner materials, particularly in terms of compatibility and sensitiveness properties, reveals no areas of concern. The use of DOA to coat the RDX (to produce CXM-7) desensitises the material to impact and friction and renders it suitable for use in explosive processing. The Vacuum Thermal Stability tests conducted on the liner materials, Rextac and Epolene N-10 display no evidence of incompatibility with each other or with PBXN-109.

Examination of the two solid ingredients, RDX (CXM-7) and aluminium, by the Malvern Mastersizer and the Scanning Electron Microscope, shows both materials have a fairly large particle size distribution. The ratio of mean particle sizes for RDX / Al is approximately 10 and thus conducive to producing a good packing efficiency and thus a low end-of-mix viscosity for the PBXN-109 slurry. The aluminium particles display a high degree of spheroidicity, also conducive to low viscosities, while the RDX has an angular and plate-like structure that is counter-productive to optimising the viscosity.

4.2 Processing Studies

The PBX was found to process well and while viscometry readings could not be recorded visual observation showed the mix to possess an acceptable although higher than preferred viscosity. It was also observed that some of the small-scale mixes appeared to exhibit different end-of-mix viscosities as evidenced by a varying degree of difficulty in casting PBXN-109 into small moulds. This was attributed to small changes in the overall plasticiser content brought about by variation of the CXM-7 composition within its 25 kg capacity container. Analysis of samples taken from various positions within the container displayed a range of plasticiser content but due to the small sample sizes of CXM-7 used in most of the mixes an average value was used in determining ingredient levels.

In general, casting of PBXN-109 into moulds (boxes or tubes) was satisfactorily carried out with little or no porosity (as evidenced by X-ray or dissection). However, during protracted casting operations difficulty in obtaining good quality castings was sometimes experienced even with the careful application of vacuum and vibration. This instigated an investigation into means of improving the processing properties of PBXN-109 by changes to the RDX particle size ratio, this lead to an expanded area of work that has been reported separately [5]. A significant improvement was achieved by the addition of small amounts of RDX Class 5 (fine) to RDX Grade A, Class 1 (coarse). The effect of increasing the level of fine RDX is shown clearly in Figure 11. Also contributing to the lower viscosity is the morphology of the RDX Grade A, Class 1. The particles exhibit a rounded shape that is preferable for processing in contrast to the angular and plate-like structure of the RDX in CXM-7. During this investigation it was interesting to note that incorporating the cure catalyst, TPB, in a master batch of binder ingredients prompted an immediate reaction between the hydroxyl and the isocyanate functional groups upon addition of the IPDI, thus greatly reducing the pot life. Under normal mixing conditions the cure catalyst has an initial latent action thus contributing to a long pot life before the cure reaction proceeds at an accelerated rate.
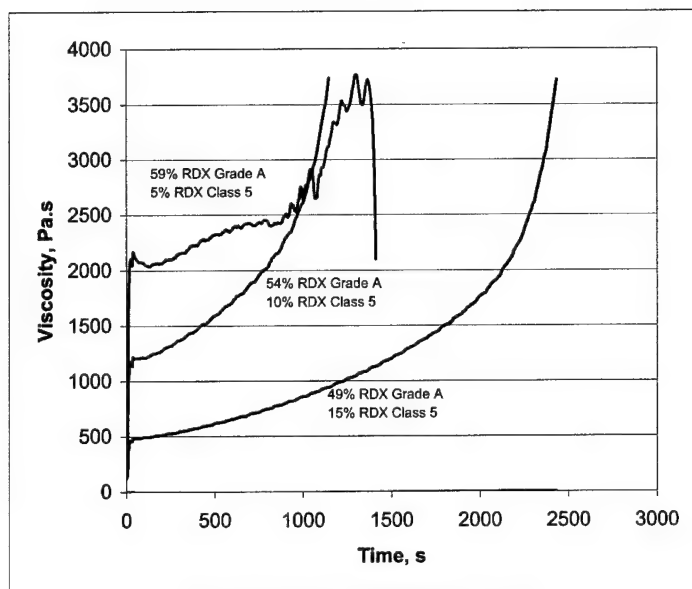
Figure 11 Effect of RDX particle size distribution on viscosity of PBXN-109

4.3 Characterisation of PBXN-109

Sensitiveness testing of the cured explosive showed that PBXN-109 was not particularly sensitive to the range of tests recorded in Table 3. Results from other sources that use comparable test equipment were not available although impact, friction and ESD testing on alternative equipment showed the same relative levels of insensitivity referenced to Composition B and TNT [12]. The Vacuum Thermal Stability test performed on a sample of PBXN-109 and the liner material revealed no signs of material incompatibility. The recorded value of 0.08 mL.g^{-1} is well below the 2.0 mL.g^{-1} maximum value permitted in reference [13]. While these results were expected it was reassuring to know that there are no particular concerns to handling and using this explosive or devices in which it is contained.

The scatter in the density values from a number of small-scale mixes is not unexpected knowing that small changes in the composition are likely to occur (eg. fluctuations in CXM-7 composition). However, the density of the PBXN-109 taken from various locations within a cylindrical casting is more consistent and reflects the larger batch size and the use of a single source batch. The result, 1.656±0.005 g.cm^{-3}, differs slightly from the density given in [12] (1.71 g.cm^{-3} with a TMD 1.752 g.cm^{-3}, i.e. 97.6%TMD) although the 98.98% correlation with TMD (1.673 g.cm^{-3}) is good. It is interesting to note that there is no apparent segregation of solids during cure.

The simplistic test used to reveal any potential problems due to the loss of plasticiser through migration to the free surface and volatilisation did show a tendency for a small quantity of liquid binder material to migrate to the free surface. No attempt was made to

analyse the material that had migrated to the surface but it was evident from its tacky nature that it was not solely DOA but also contained other binder ingredients. Prolonged storage at 60°C did not result in a loss of DOA as evidenced by a mass loss. The reason for the appearance of this material at the surface is not clear as it was not an occurrence exhibited by all castings of PBXN-109 and did not appear to be influenced by the degree of vibration during casting. A study [12] to examine the change in density of PBXN-109 as a function of storage time and temperature corroborated this finding. Under the most severe conditions examined, 8 weeks at 160°F (71°C), no change in density was observed.

The Military Specification for PBXN-109 [3] requires the cured explosive to have a stress (maximum) at 25°C no less than 60 psi (0.41 MPa) and a strain at maximum stress at 25°C greater than 12%. For the smaller scale mixes (0.5 kg and 6 kg) the strain data presented in Table 7 does not comply with this requirement. However, for a production size batch of PBXN-109 the mechanical properties criteria are easily met. This highlights size effects and the need to consider scale-up factors when developing formulations for production size mixes. Mechanical property data for unaged PBXN-109 taken from other sources reveals that a considerable range in properties can be expected. Data generated by Royal Ordnance (Glascoed) [14] shows the maximum tensile strength as 0.48 MPa, the strain at maximum stress as 36% and the modulus of elasticity as 2.23 MPa. While some differences are attributable to batch-to-batch variations of the ingredients employed in the formulation, particularly the HTPB and CXM-7, large differences can result from the choice of curing conditions to which the PBX is subjected. Mil-E-82886(OS) [3] and NMD 161D [13] requires that PBXN-109 be cured at a temperature between 30°C and 60°C until a minimum hardness of 30 Shore A is attained. The samples manufactured and tested by WSD/DSTO were cured at 60°C (nominal) for 7 days thus ensuring a complete cure. These conditions tend to maximise the stress value and minimise the strain value.

Comparison of the properties of the two 0.5 kg mixes presented in Table 7 does suggest that a small compositional difference does exist although it must be recognised that the difference in using pre-coated and uncoated RDX may also have an affect.

The data presented in Table 8, particularly for strain and modulus, show a distinct trend for the properties to change with progression down the mould. The statistical data also supports this, while three specimens is a small test population it can be seen that the property standard deviation within a slab (from which the three specimens are taken) is markedly less than the standard deviation of all specimens (21) taken from the block. This is in contrast to the density data (discussed above) that indicate no apparent change in values within a cylindrical casting. The reason for this discrepancy is unknown but may relate to the viscosity of the PBX slurry at the time of casting. Unfortunately, density measurements were not conducted on samples taken from the slabs used to cut out mechanical property specimens.

The tensile properties displayed as a function of temperature in Table 9 show that the PBX possesses good mechanical properties over a considerable temperature range. It is not until the test temperature falls below –40°C that a dramatic change in mechanical

properties occurs. The strain data from Table 9 is reproduced in Figure 12 showing the glass transition temperature, T_g, for this PBX lies below –50°C. Additional data at low temperatures is required to more accurately determine the T_g of this system. In [12] the T_g is quoted as –88°C but the method of determination was by linear thermal expansion and hence not directly comparable. Below the glass transition temperature the PBX will become hard and brittle and its sensitivity to unplanned stimuli may increase. However, the value measured for this property is not regarded as a problem (see Section 4.6 for further comment on this topic).
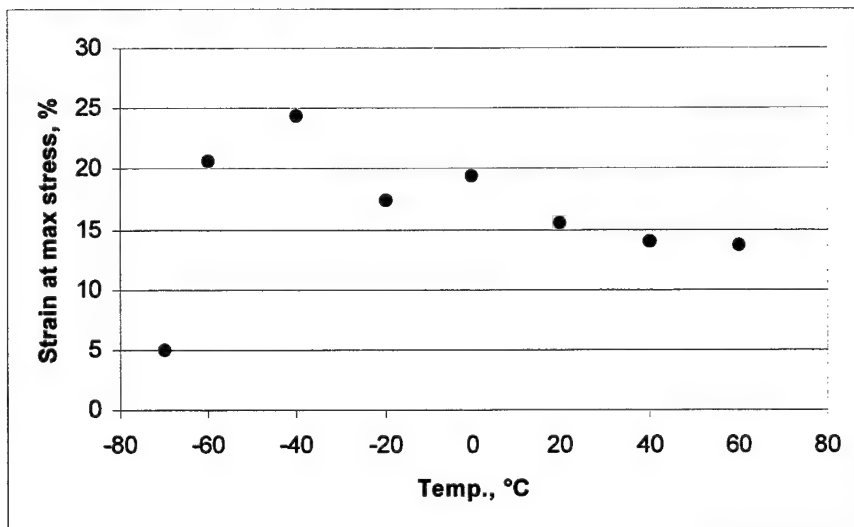

Figure 12 The strain at maximum stress as a function of temperature for PBXN-109

The nature and properties of the materials used to perform a Bond-in-Tension test between the liner material and PBXN-109 precluded this test being satisfactorily conducted. However, it was apparent that adhesion between the two materials was poor and the bond easily disrupted. While a good bond strength between the liner and explosive may not be essential it was considered a potential problem in light of the shrinkage exhibited by the explosive during cure and post cure cool down. A means of determining the linear and volumetric contraction of the PBX was not available and the magnitude of the problem could not be determined. However, qualification tests [15] performed on the first batch of warheads produced by ADI Ltd showed the dimensional changes were sufficient to permit movement of the PBX fill during vibration testing.

4.4 Explosive Properties

Due to the problems associated with the accuracy of the digital camera records the results for the 102 mm confined charge (coaxial probes, 7579 m.s^{-1}) and the 82 mm unconfined charge (piezoelectric pins, 7617 m.s^{-1}) are considered more representative of the actual values. The values quoted in the literature [16] for PBXN-109 are 7602 m.s^{-1} (ρ=1.681g.cm^{-3})

and 7630 m.s^{-1} (ρ=1.697g.cm^{-3}) are comparable to the values determined in this study. The differences are probably insignificant and attributable to the accuracy of the measuring technique and variations in test specimen density.

The relative detonation pressure shown in Table 11 is lower than the 23.7 GPa (ρ=1.681g.cm^{-3}) value reported in the literature [16]. The difference is more likely a consequence of the measuring techniques rather than a property of the explosive. Rather than providing a direct measurement of detonation pressure the Dent Test operates by comparison with a known standard and is thus subject to a certain degree of error.

At the time of conducting this work a suitable method of casting acceptable quality cylindrical charges with a diameter less than 10 mm was not available. Consequently, a result for a charge less than 10 mm diameter was not obtained. Since the 10 mm charge detonated the critical diameter for PBXN-109 is less than 10 mm which is consistent with the value of 7 mm reported in the literature [16].

4.5 Insensitive Munitions Characterisation

The small-scale cook-off test was developed to provide information additional to that derived from standard laboratory-scale thermal tests eg. Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA) and Thermal Gravimetric Analysis (TGA). By heating a larger test sample, in this case about 20g, under some degree of confinement an estimate of the degree of violence of the resulting reaction can be made. While this test was not performed to indicate how a Penguin warhead would necessarily respond to cook-off it was intended to highlight any potential problems and provide baseline data for the ageing trial. It should be noted that the selected heating rates differ from those listed in MIL-STD-2105B [17] for cook-off tests on items of ordnance particularly for slow cook-off where a heating rate of 3.3°C.hr^{-1}is required. For laboratory testing a faster heating rate and hence shorter testing time is deemed appropriate.

The mild reaction exhibited by all samples tested is similar to the findings of other investigators [12, 18]. The size and geometry of the sample, the heating rate, and the use of an elevated temperature pre-soak govern the time to reaction and reaction initiation temperature [12]. The data in Table 12 shows a fair degree of variability and with the small number of tests performed does not permit a statistical analysis. However, in general it appears that the fast heating rate tests react at a slightly higher temperature than the slow rate tests. Previous work [10] using this form of test equipment has demonstrated similar results for a range of different explosive compositions. The results at both heating rates have been corrected by means of calibration tests to provide the temperature of the explosives surface rather than the indicated thermocouple reading. The temperature difference is thus predominantly a consequence of time, thermal conductivity and degradation kinetics. The data indicates also that the presence of the wax liner on the tubes increases the temperature at which a reaction occurs. Although the increase is small and may be due to test-to-test variability it may be attributable to the thin layer of wax between the tube and the explosive providing an insulating effect.

The LSGT provides an indication of the shock sensitivity of PBXN-109 but does not quantify the shock wave that will initiate the Penguin warhead. The data serves as an indication of ranking against other explosive compositions tested in a similar manner and forms the baseline data against which aged material can be compared.

The data in Table 13, 196 cards and 2.35 GPa, is the same for lined and unlined tubes indicating no effect from the wax. In a US study [19] to investigate the effect of RDX composition variability on the shock sensitivity of PBXN-109 typical results were in the range 192 to 204 cards (2.28 GPa to 1.98 GPa). These results are in reasonable agreement with the present work, the differences being due to the nature and particle size distribution of the RDX and slight differences in test methodology.

4.6 Ageing Characteristics

With the limited mechanical property data available for aged samples of PBXN-109 it is difficult to determine the exact nature of the trend other than to say that there does not appear to be a reason for concern. The explosives stored for 12 months under ambient conditions (Table 14) do not show a significant trend. The variation in properties may be attributable to slight composition changes in the test specimen as a consequence of the position from which it was taken from within the casting mould. The material aged at 60°C (Table 14) does indicate a slight change in mechanical properties, particularly for the exposed material with an increase in stress and modulus.

Comparing the two sets of data presented in Table 15 indicates a difference between the PBX aged at RT and that aged at 60°C for the strain recorded at maximum stress as a function of temperature (see Figure 13). It is also noted that below -20°C there is a discrepancy in the modulus of elasticity data for these two ageing conditions. The data suggests that for the PBX aged at 60°C the T_g has been raised from approximately -50°C to -20°C. The reason for such a change can only be speculated and is beyond the scope of this report. However, it may be prudent to investigate this low temperature effect at some later date since such a change may have an adverse effect on response to external stimuli such as bullet and fragment impact.
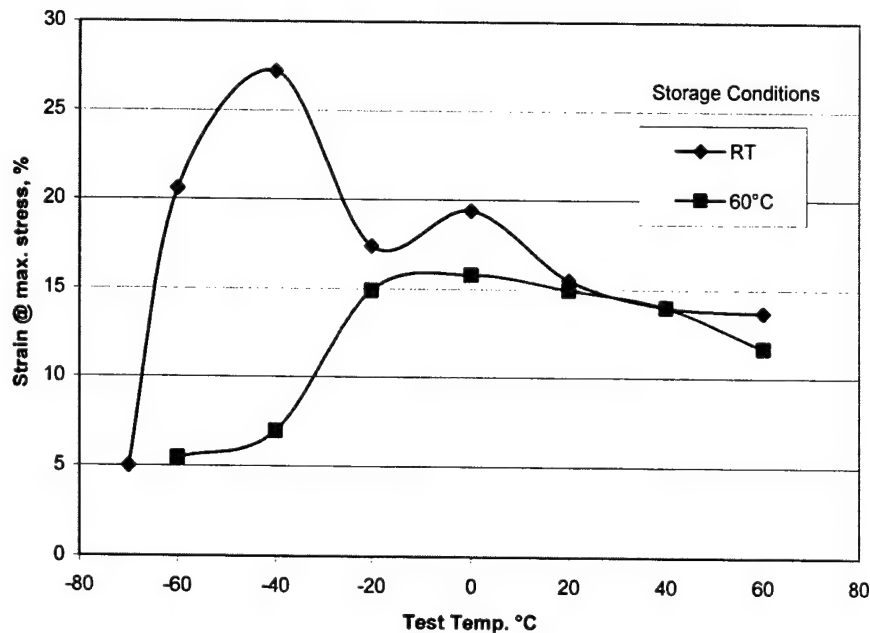
Figure 13 Comparison of strain data for aged PBXN-109

In a comprehensive ageing trial of PBXN-109 in which changes were monitored by mechanical property measurements [12] samples were stored as slabs wrapped in Al foil placed in 77, 120, 140 and 160°F ovens. Samples were aged for 1, 2, 4 and 8 weeks. The samples were tested in a manner similar to the present study. The results show a considerable change in stress, strain and modulus particularly for the elevated temperature samples. The effect is most pronounced during the first two weeks after which the rate of change is considerably less. The early large changes in properties are probably due to post cure reactions and because of the magnitude of the changes it would indicate that the original material was not fully cured. The values extracted from the ageing curves presented in [12] for the initial mechanical properties and those after 8 weeks ageing at 140°F (60°C) are shown below in Table 19 which includes data from this study for comparison.

Table 19 Selected ageing data from [12] and this study

	Stress (max) MPa	Strain at max stress %	Modulus of elasticity MPa
Wagenhals & Smith [12]			
Zero time	0.54	22.0	3.1
8 weeks at 60°C	0.76	15.0	9.7
This study			
Zero time	0.65	13.9	8.3
12 months at 60°C (sealed)	0.71	14.2	8.5

Comparing the two sources of aged data in Table 19 it can be seen that there is reasonable agreement in the values. However, comparison of the unaged material shows a large discrepancy in properties. In this study the original and aged material display only minor differences whereas the Naval Weapons Center data shows very large differences. It would thus appear that the PBXN-109 produced at NWC was considerably under cured and continued to cure during the ageing trial. In [12] the mechanical property data were used to determine Arrhenius kinetic reaction rates that were compiled with other thermal data and plotted as reaction rate versus inverse temperature. From these data it was concluded that there is a possible long term potential for early ageing of the PBX. It is unfortunate that the period of ageing in [12] was not extended to 6 or 12 months. The limited but longer term ageing data from this study does not support these findings. While no data are provided [19] reports that during a two year ageing trial PBXN-109 held at 40°C showed no significant changes in mechanical properties.

The investigation of the effect of ageing on IM characteristics as represented by cook-off and shock sensitivity behaviour was conducted to determine whether these properties were adversely affected during long-term ageing. Within the limits of the experimental accuracy of the data it would appear that there is no change in either cook-off (fast or slow heating rate) or shock sensitivity (Tables 16 to 18). One means by which an aged PBX may be expected to influence cook-off and shock sensitivity properties would be due to changes in binder chemistry. In becoming harder and more brittle the sensitivity of the PBX to thermal and shock loading may be expected to change. This lack of change in these properties is reflected in the correspondingly small change in mechanical properties for the same period of ageing. An ageing trial conducted at Naval Surface Warfare Center, Indian Head Division [19] used the LSGT to assess changes in shock sensitivity of PBXN-109 aged for 12 months at 60°C. In that study the initial 50% point was 197 cards, the 6 month 50% point was 212 cards and the 12 month 50% point was 211 cards. The initial value may be a consequence of the state of cure but like the present study the change is negligible.

5. Conclusions and Recommendations

The foundation of a database of properties of the ingredients used in the polymer bonded explosive PBXN-109 and of the properties of PBXN-109 has been established. Characterisation studies have not identified any materials that pose undue safety or sensitivity concerns either during handling, processing or during long-term storage. Some properties that were considered marginal included the processing properties, in particular the fairly high end-of-mix viscosity, and the mechanical properties, in particular the strain capability at maximum stress, of PBXN-109. However, both of these short-comings were either reduced or negated by the factor of scale – large-scale mixes such as those produced in a 30 gallon mixer exhibited acceptable properties.

The ageing studies addressed in this program of work were of a very limited nature and related only to the explosive as a separate entity and did not consider ageing effects displayed by PBXN-109 once filled in the Penguin warhead. The data from the shock sensitivity and cook-off tests do not indicate any problems as a consequence of long-term ageing. However, there is a degree of uncertainty concerning the possible elevation of glass transition temperature during ageing and its potential to affect shock sensitivity. It is recommended that a thorough investigation be conducted into the effects of ageing on the low temperature mechanical properties of PBXN-109 and into the shock sensitivity properties at temperatures above and below the T_g. It is also recommended that an ageing programme that embraces all environmental factors be conducted on the warhead with particular emphasis on changes in the response to shock, thermal and impact (bullet and fragment) aggression.

It has been noted that dimensional changes occurred in the first warheads filled with PBXN-109 by ADI Ltd. This was attributed to a combination of thermal and cure shrinkage but the phenomenon was unable to be investigated due to the lack of suitable instrumentation. As a decrease in the volume of the warhead explosive contents enables the charge to move during transport (land, sea, air) an investigation into potential problems and remedies is recommended.

Work at DSTO [5] has shown that the shock sensitivity of PBXN-109 can be significantly reduced by the use of a reduced sensitivity grade of RDX such as that produced by ADI Ltd or SNPE. It is known that the use of this less sensitive explosive fill alone would not suffice for the warhead to pass the sympathetic detonation criteria. However, by combining the use of a reduced shock sensitive version of PBXN-109 with advanced packaging and mitigation techniques it should be possible to produce a warhead that complies with IM sympathetic detonation criteria.

6. Acknowledgements

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4. AUTHOR(S)	5. CORPORATE AUTHOR
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6a. DSTO NUMBER DSTO-TR-1471	6b. AR NUMBER AR-012-847	6c. TYPE OF REPORT Technical Report	7. DOCUMENT DATE August 2003

8. FILE NUMBER E9505-25-82-1	9. TASK NUMBER NAV 98/079	10. TASK SPONSOR DNAPO	11. NO. OF PAGES 30	12. NO. OF REFERENCES 19

13. URL on the World Wide Web	14. RELEASE AUTHORITY
http://www.dsto.defence.gov.au/corporate/reports/DSTO-TR-1471.pdf	Chief, Weapons Systems Division

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18. DEFTEST DESCRIPTORS
Polymer Bonded Explosives, Anti-Ship Missile, Insensitive Munitions, Ageing (materials), Performance Evaluation, Performance Tests

19. ABSTRACT

The RAN has acquired the Penguin Anti-Ship Missile for deployment from Kaman SH-2G(A) Super Seasprite helicopters which are to be carried by the ANZAC frigates. The Penguin manufacturer, Kongsberg Defence and Aerospace of Norway, negotiated an industry off-set program with local industry, a component of which was for ADI Ltd to fill the missile warhead with the polymer bonded explosive PBXN-109. This was the first example of Australian industry production of a PBX for service use. DSTO was tasked to provide appropriate local advice as required to the Penguin Project Office and ADI Ltd during introduction into service and onwards through life. This report details the support to this programme and the the results of work to establish a database on processing, chemical and mechanical properties, performance, and hazards response of PBXN-109.